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Concentration of camu–camu juice by the coupling of reverse osmosis and osmotic evaporation processes

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A B S T R A C T

The objective of this work was to evaluate the technical feasibility of coupling two membrane separation processes, reverse osmosis (RO) and osmotic evaporation (OE), in order to concentrate clarified camu–camu juice, focusing on the vitamin C, phenolic compounds and antioxidant activity of the final product. The juice was firstly pre-concentrated by RO, reaching 285 g kg^{-1} of soluble solids. During this step, the juice's osmotic pressure showed to be the main factor controlling mass transfer. The juice was then concentrated by OE, reaching 530 g kg^{-1} of soluble solids. Vitamin C, total phenolics and antioxidant activity levels of 94.6 g ascorbic acid kg^{-1}, 105.2 g galic acid kg^{-1} and 762 mmol Trolox kg^{-1}, respectively, were achieved in the final product. The use of integrated membrane processes proved to be an interesting alternative to the concentration of thermosensitive juices, reaching concentration levels up to 7 times for camu–camu juice's bioactive compounds.

1. Introduction

Camu–camu (*Myrciaria dubia* (*H.B.K.*) *Mc Vaugh*) is a fruit from the Amazonian region, found on the margins of rivers and lakes. Its main characteristic is the high vitamin C content, with reported values ranging from 1000 to 6000 mg/100 g (Chirinos et al., 2010; Justi et al., 2000). Camu–camu is also considered a good source of polyphenols, with values higher than 1000 mg galic acid/100 g (Maeda et al., 2006; Rufino et al., 2010; Zanatta et al., 2005). Its high phenolic content, together with vitamin C, contributes to its high antioxidant capacity and consequent health benefits.

The high concentration of ascorbic acid and phenolic compounds results in the high acidity of camu–camu, which does not attract the consumption of its fresh pulp. However it can be mixed to other fruits in order to provide a nutritional enrichment, besides serving as raw material for obtaining products such as ice cream, nectars, jams and yoghurt (Rodrigues et al., 2004). With the development of increasingly globalized markets, the need of reduced costs associated to logistics operation (packaging, storage and transportation) has become a fundamental point for products competitiveness and conquest of new markets. In this sense, the concentration processes stand out as an important tool to facilitate commercialization, especially for imports and exports.

In general, fruit juices are preserved and concentrated by thermal processes such as pasteurization and vacuum evaporation. However, the product heating during these processes can change the natural aroma and flavor of the fresh juice and cause degradation of thermosensitive compounds such as vitamin C and other bioactive compounds responsible for its antioxidant activity (Cassano et al., 2007; Fernandes et al., 2007; Galaverna et al., 2008).

Membrane technology is an alternative to the conventional processes for juice concentration and clarification (Álvarez et al., 2000; Girard and Fukumoto, 2000). It has many advantages over traditional separation processes: in general separation occurs at room temperature, with no phase change and without using a heat source, resulting in considerable energy savings and avoiding oxidation and degradation of thermolabile compounds (Mulder, 1996). Among the different techniques of membrane separation, reverse osmosis and osmotic evaporation have stood out for their potential for concentration of fruit juices (Girard and Fukumoto, 2000; Vaillant et al., 2001).

Juices concentration by reverse osmosis has been evaluated for temperate and tropical fruits, showing satisfactory results regarding the preservation of the final product quality (Cassano et al., 2003; Kozák et al., 2008; Jesus et al., 2007). Reverse osmosis (RO)

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is a process in which a hydraulic pressure greater than the solution osmotic pressure is applied, so that water permeates from a high to low solute concentration. However, this pressure driven process is limited by the product osmotic pressure that increases with increasing concentration. For this reason, RO is in general considered a pre-concentration technique, allowing juice concentration only up to 25–35°Brix (Couto et al., 2011; Jesus et al., 2007).

Osmotic evaporation (OE) is a process able to remove water from a solution at low temperature and pressure. The driving force is the concentration difference between the solution being concentrated (in this case, the juice) and a hypertonic solution, typically a concentrated brine (Vaillant et al., 2001). Juices concentrated by osmotic evaporation can achieve high soluble solids content, higher than 60 Brix, keeping their nutritional characteristics (Cassano et al. 2003; Cissé et al., 2011; Koroknai et al., 2006).

However, the use of osmotic evaporation as a direct process to concentrate fruit juices is difficult to implement at industrial scale, since the large amount of water present in the initial juice promotes a fast brine dilution, which negatively affects the water removal from the juice. In this context, the coupling of reverse osmosis and osmotic evaporation can be considered a promising alternative, since it results in products with similar solid content than those obtained by conventional methods (such as vacuum evaporation), with less pronounced effects on the juice's quality.

The objective of this work was to evaluate the effect of coupling reverse osmosis and osmotic evaporation processes on the quality of concentrated camu–camu juice. The processes were evaluated in terms of permeate flux and volumetric reduction ratio, and the juices were evaluated for total and soluble solids, total acidity, ascorbic acid, total phenolic content and antioxidant activity.

2. Materials and methods

Fig. 1 illustrates all the steps performed for camu–camu juice concentration.

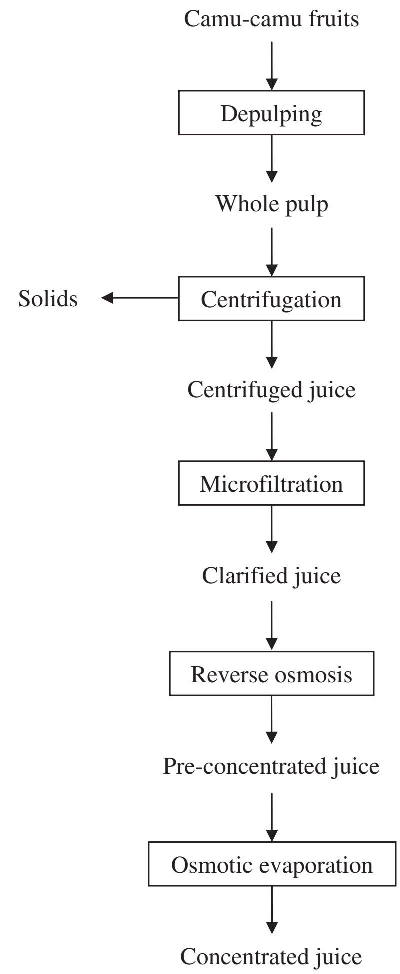

Fig. 1. Flux diagram of camu–camu juice concentration by coupling reverse osmosis and osmotic evaporation processes.

2.1. Raw material and clarified juice processing

Frozen camu–camu pulp was acquired at the Central Supply Market of Rio de Janeiro state. The pulp was thawed according to the amount necessary for each process. The camu–camu pulp was initially centrifuged, aiming to standardize and reduce the suspended solid content, with the aid of a basket centrifuge SIZE 2 (International Equipment Company, Needham, USA), at 4000 rpm (479.2 g), using a 150 μm Nylon® screen as the filter media. The centrifuged juice was then clarified in a semi-pilot system of crossflow microfiltration (TIA, Techniques Industrielles Appliquées, Bollene, France) consisting of four tubular ceramic membranes of α-alumina, T1-70 (Pall Corporation: Membralox® Ceramic Membrane Products, New York, USA), with mean pore size diameter of 0.1 μm and total permeation area of 0.022 m^2. Microfiltration was performed at 45 °C, with a transmembrane pressure of 2.5 bar and tangential velocity of 6.9 m/s.

2.2. Concentration

The concentration of camu–camu juice was carried out in two steps. Firstly, the clarified juice was concentrated by reverse osmosis up to around 300 g kg^{-1}. Then, this juice was further concentrated by osmotic evaporation up to 550–600 g kg^{-1}.

2.2.1. Reverse osmosis

The pre-concentration of the clarified juice by reverse osmosis was performed in a plate and frame reverse osmosis system Lab Unit 20 (DSS, Silkeborg, Denmark), composed of HR98PP thin film composite membranes (DSS, Silkeborg, Denmark), with 98% nominal rejection to a 0.25% NaCl solution and permeation area of 0.288 m^2. The process was carried at 20 °C and 60 bar, according to Rodrigues et al. (2004).

2.2.2. Osmotic evaporation

Osmotic evaporation was carried out in batch mode in a lab scale system consisted by two independent circuits, one for the juice and the other for the brine. The hydrophobic flat sheet membrane (Pall Gelman – TF 200) with effective surface area of 0.032 m^2 was located in the middle of a stainless steel cell (Fig. 2). This membrane is composed by a thin polytetrafluoroethylene selective layer supported by a polypropylene macro porous layer. According to the manufacturer, its average characteristics are 60% porosity, 0.2 μm average pore diameter and 165 μm thickness. Approximately one liter of 5.5 M $CaCl_2$ solution was used as brine. The brine and the pre-concentrated juice were kept at 20 °C and 35 °C using thermostatic baths, with maximum transmembrane pressure of 0.2 bar in order to avoid aqueous linkages through the membrane. During osmotic evaporation, the brine and the juice were kept under circulation at a flow rate of approximately 80 kg h^{-1} and $CaCl_2$ crystals were added to maintain the brine solution near saturation (at 5.5 mol L^{-1}).

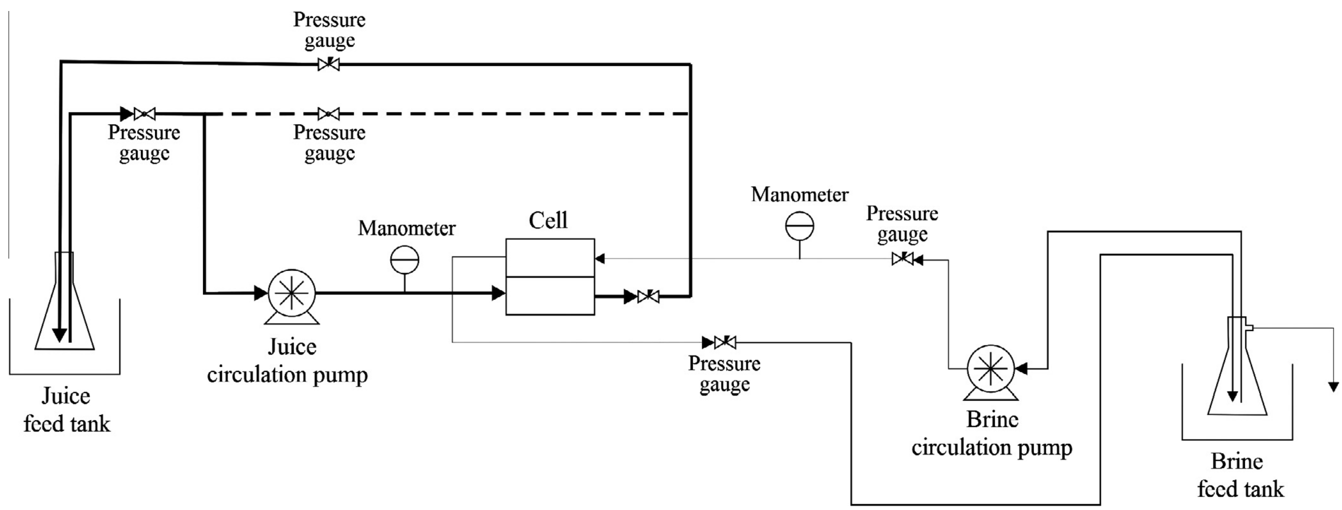

Fig. 2. Schematic representation of the osmotic evaporation system.

2.3. Process evaluation

The concentration processes were evaluated with respect to permeate flux and volumetric reduction ratio (VRR), which were calculated according to the following equations:

$$J = \frac{V}{A \times t} \tag{1}$$

$$\text{VRR} = \frac{V_C}{V_F} \tag{2}$$

where V is the permeated volume in a time t, A is the membrane surface area, V_C is the final volume of concentrate and V_F is the initial feed volume.The concentration factor (CF), defined as the concentration of the component i in concentrate ($X_{i,C}$) and feed ($X_{i,F}$) fractions was also calculated (Eq. (3)).

$$\text{CF} = \frac{X_{i,C}}{X_{i,F}} \tag{3}$$

2.4. Physicochemical characterization

The clarified camu–camu juice and the products concentrated by RO and OE were evaluated for their main chemical and physical parameters.

Samples were analyzed for pH, total and soluble solids, total acidity and ascorbic acid according to A.O.A.C. (2006). Total phenolic content was determined with the Folin–Ciocalteu reagent according to the method described by Georgé et al. (2005), being expressed as g gallic acid per kg of juice. Antioxidant activity was measured by the Trolox Equivalent Antioxidant Capacity (TEAC), also known as ABTS cationic radical scavenging activity (Re et al., 1999), being expressed as mmol Trolox per kg of juice.

Analyses were performed in triplicate and the results were evaluated statistically by Tukey test at 5% significance level ($p < 0.05$) with the software XLSTAT pro 7.5®.

3. Results and discussion

3.1. Reverse osmosis

Fig. 3 shows the evolution of the permeate flux and soluble solids during camu–camu juice pre-concentration by reverse osmosis. There was a decrease in the permeate flux and an increase in the soluble solids concentration along time. This flux decrease may be attributed to the increase in the osmotic pressure that decreases the driving force and in the juice viscosity, which occurs due to the increase of solids content during water removal, making the mass transfer more difficult. The average permeate flux (calculated as the mean value between the fluxes observed overtime) was 11.3 L h^{-1} m^{-2} and the volumetric reduction ratio achieved at the end of the process was 4.2.

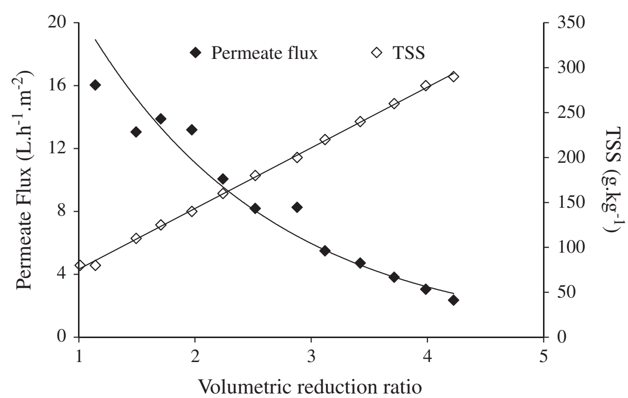

Fig. 3. Permeate flux and soluble solids content as a function of the volumetric reduction ratio in the pre-concentration of camu–camu juice by reverse osmosis.

Concentration by reverse osmosis is also limited by the membrane fouling and the concentration polarization layer. This last factor has special importance due to the increase in osmotic pressure as the concentration at the membrane surface increases.

In membrane processes governed by a pressure gradient, the permeate flux can be expressed by a phenomenological equation like:

$$J_P = K \times \Delta P \tag{4}$$

where K and ΔP are, respectively, the overall mass transfer coefficient and the pressure difference between the two sides of the membrane.

ΔP is the difference between the hydraulic pressure (P) and the osmotic pressure (π) in the two sides of the membrane, feed (f) and permeate (p):

$$\Delta P = (P_f - \pi_f) - (P_p - \pi_p) \tag{5}$$

$$\Delta P = (P_f - P_p) - (\pi_f - \pi_p) \tag{6}$$

In the case of reverse osmosis, considering that the permeate is composed mostly by water, the osmotic pressure in this fraction can be approximated to zero, and the difference ($P_f - P_p$) is the hydraulic pressure applied to the system (P_{hydr}), which is constant. Thus, Eq. (4) can be expressed as:

$$J_P = K \times (P_{hydr} - \Delta\pi) \tag{7}$$

$$K = \frac{J_P}{(P_{hydr} - \Delta\pi)} \tag{8}$$

According to these definitions, the plot of $K \times$ VRR can be used to estimate if the changes in the permeate fluxes are mostly due to the increase on the osmotic pressure or to other factors like membrane fouling, concentration polarization or the increase of juice's viscosity. According to Thijssen (1970), the osmotic pressure (π, bar) can be calculated as a function of the soluble solids content (SS, g kg^{-1}), as follows:

$$\pi = \frac{133.75 \times SS}{1000 - SS} \tag{9}$$

From Eq. (9), it is possible to determine the evolution of the osmotic pressure of camu–camu juice along of the reverse osmosis process, as illustrated in Fig. 4.

Similarly to the soluble solids content, the osmotic pressure increased linearly with the volumetric reduction rate, until reaching approximately 50 bar. At this point, the osmotic pressure is relatively close to the hydraulic pressure (60 bar), which can be one of the reasons of the permeate flux reduction.

However, in order to verify if some factors other than the osmotic pressure had affected the permeate flux, Fig. 5 was plotted, based on Eq. (8).

According to Fig. 5, when the juice was concentrated by reverse osmosis, the overall mass transfer coefficient reached values around 0.3 L h^{-1} m^{-2} bar^{-1} and did not considerably vary along the process. This indicates that the permeate flux decreased in the same proportion that the osmotic pressure increased, suggesting that this factor was the main responsible for the flux reduction.

The characterization of both the clarified camu–camu juice (feed of reverse osmosis process) and the juice concentrated by reverse osmosis is shown in Table 1.

Total solids content increased from 75 to 288 g kg^{-1}, representing a concentration degree of 3.84. This result is similar to that observed by Rodrigues et al. (2004) during the reverse osmosis concentration of camu–camu juice clarified by the association of an enzymatic hydrolysis and microfiltration. The total titrable acidity and soluble solid content increased proportionally to the total solids concentration.

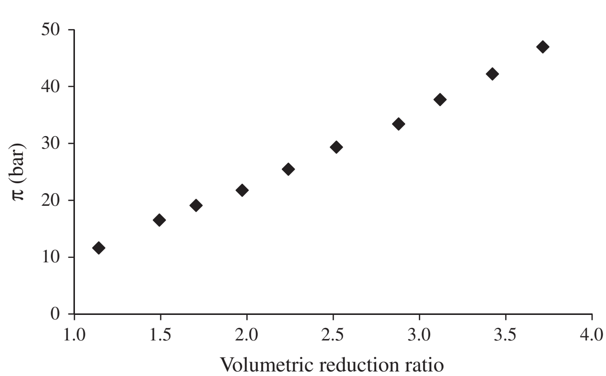

Fig. 4. Osmotic pressure as a function of the volumetric reduction ratio during the pre-concentration of camu–camu juice by reverse osmosis.

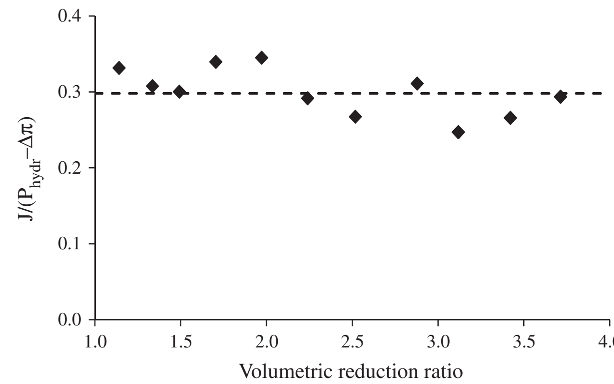

Fig. 5. $J/(P_{hydr} - \Delta\pi)$ as a function of the volumetric reduction factor, during the pre-concentration of camu–camu juice by reverse osmosis.

Vitamin C, total phenolics and antioxidant capacity increased 3.3, 3.8 and 4.2 times, respectively. The determined values of total phenolic compounds in the clarified and in the concentrated camu–camu juice indicate that there were no significant losses of these phytochemical compounds during reverse osmosis. Considering the results expressed in a dry basis, in order to eliminate the concentration effects, around 13% of vitamin C was lost during processing. This loss may be related to some oxidation occurring during concentration or to the passage of some compounds through the reverse osmosis membrane. Rodrigues et al. (2004) observed slightly lower losses (7.6%) in the reverse osmosis of camu–camu juice previously clarified by an enzymatic treatment associated with microfiltration.

On the other hand, the antioxidant activity of the concentrated juice increased in a higher degree as compared to the vitamin C and phenolic content. This may indicate that some compounds present in the camu–camu juice, other than vitamin C and phenolics, which also have antioxidant properties, may have been concentrated during processing.

Aguiar et al. (2012) observed phenolic losses around 11% and antioxidant activity reduction of 21% in the concentration of clarified apple juice by reverse osmosis. Bogianchini et al. (2011) evaluated the influence of reverse osmosis process on the bioactive compounds concentration of dealcoholised wine. The authors observed that the phenolic levels and the antioxidant activity were well preserved during the process.

3.2. Osmotic evaporation

The permeate flux during camu–camu concentration by osmotic evaporation is shown in Fig. 6. A volumetric reduction ratio close to 3.0 was achieved. The permeate flux varied from 3.3 kg h^{-1} m^{-2}, at the beginning of process, to 0.2 kg h^{-1} m^{-2}, when total solids reached 550 g kg^{-1}. Permeate fluxes between 2 and 13 kg h^{-1} m^{-2} were obtained by Hongvaleerat et al. (2008) in the concentration of pineapple juice by osmotic evaporation, using the same membrane of the present work. Courel et al. (2000) observed fluxes varying between 0.5 and 23 kg h^{-1} m^{-2}, by varying operating parameters such as solute content, circulation velocity and temperature of the fluids, also using a TF200 membrane to concentrate sucrose solutions.

The increase in total solids promotes the increase in the juice viscosity, which is also one of the causes for the reduction of the permeate flux (Hongvaleerat et al., 2008). In addition, the increase in solids content results in a reduction in the juice's water activity, i.e., a reduction in the partial vapor pressure gradient across the membrane, which is the driving force for water mass transfer.

Table 1
Physicochemical characteristics of camu–camu juice pre-concentrated by reverse osmosis.

Analysis	Feed (clarified juice)	Pre-concentrated juice (RO)	Concentration factor
pH	2.9 ± 0.1[a]	2.8 ± 0.1[a]	–
Soluble solids (g kg^{-1})	73 ± 1[a]	285 ± 2[b]	3.90
Total solids (g kg^{-1})	75 ± 1[a]	288 ± 1[b]	3.84
Titratable acidity (g kg^{-1})	22.0 ± 0.5[a]	84.0 ± 0.8[b]	3.82
(g kg^{-1} dry matter)	293.3 ± 6.7[a]	291.7 ± 2.8[a]	
Vitamin C (g AA kg^{-1})	15.9 ± 0.3[a]	52.9 ± 0.8[b]	3.33
(g AA kg^{-1} dry matter)	212.0 ± 4.0[b]	183.7 ± 2.8[a]	
Total phenolics (g GA kg^{-1})	14.8 ± 0.1[a]	56.4 ± 1.7[b]	3.81
(g GA kg^{-1} dry matter)	197.3 ± 1.3[a]	195.8 ± 5.9[a]	
Antioxidant activity (mmol TE kg^{-1})	104.7 ± 4.3[a]	440.6 ± 46.0[b]	4.21
(mmol TE kg^{-1} dry matter)	1396.0 ± 57.3[a]	1529.9 ± 159.7[b]	

Different letters indicate significant difference between different samples ($p \leqslant 0.05$).

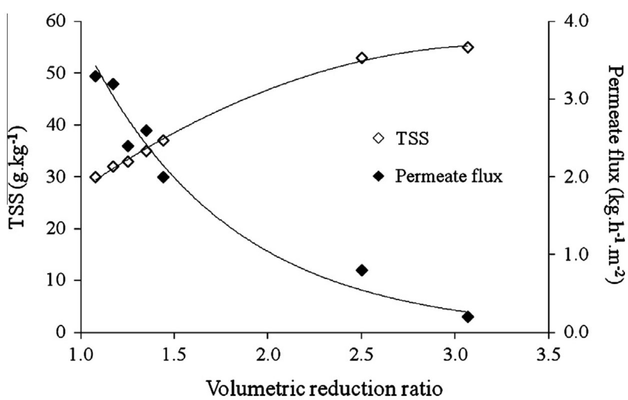

Fig. 6. Permeate flux and total soluble solids (TSS) along the concentration of camu–camu juice by osmotic evaporation.

Total solids content increased from 288 to 566 g kg^{-1}, corresponding to a concentration degree of 1.97 times. This result is similar to that found by Hongvaleerat et al. (2008) in the concentration of clarified pineapple juice by osmotic evaporation. Galaverna et al. (2008) achieved 60 Brix during the concentration of blood orange juice by osmotic evaporation, also with a concentration degree of approximately 2.

The characterization of camu–camu juice concentrated by osmotic evaporation is shown in Table 2.

Although total phenolics content have shown a slightly lower concentration degree, when compared to total solids, no significant differences were observed when results were expressed in a dry basis. It indicates that there were no losses of these compounds during processing.

Vitamin C was concentrated 1.79 times and a loss of around 9% was observed during osmotic evaporation. According to Odriozola-Serrano et al. (2007), vitamin C losses during processing occur due to the fact that it is a bioactive substance present in camu–camu very sensitive to environmental conditions such as exposure to oxygen, temperature and light. So, these reductions may be attributed to the possible occurrence of oxidative reactions, since the process was carried out for a long time (22 h), due to the small membrane surface. Rodrigues et al. (2004) evaluated the concentration of camu–camu juice by performing osmotic evaporation in two stages: in the first one, the original clarified juice was concentrated up to

Table 2
Physicochemical characteristics of camu–camu juice concentrated by osmotic evaporation.

Analysis	Feed (RO juice)	Concentrated juice (OE)	Concentration degree
pH	2.8 ± 0.1[b]	2.5 ± 0.1[a]	–
Soluble solids (g kg^{-1})	265 ± 2[a]	530 ± 2[b]	2.00
Total solids (g kg^{-1})	288 ± 1[a]	566 ± 1[b]	1.97
Titratable acidity (g kg^{-1})	84.0 ± 0.8[a]	161.0 ± 2.5[b]	1.92
(g kg^{-1} dry matter)	291.7 ± 2.8[b]	284.5 ± 4.4[a]	
Vitamin C (g AA kg^{-1})	52.9 ± 0.8[a]	94.6 ± 0.7[b]	1.79
(g AA kg^{-1} dry matter)	183.7 ± 2.8[b]	167.1 ± 1.2[a]	
Total phenolics (g GA kg^{-1})	56.4 ± 1.7[a]	105.2 ± 2.9[b]	1.87
(g GA kg^{-1} dry matter)	195.8 ± 5.9[a]	185.9 ± 5.1[a]	
Antioxidant activity (mmol TE kg^{-1})	440.6 ± 6.9[a]	762.2 ± 17.1[b]	1.73
(mmol TE kg^{-1} dry matter)	1529.9 ± 23.9[b]	1346.6 ± 30.2[a]	

Different letters indicate significant difference between different samples ($p \leqslant 0.05$).

250 g kg^{-1} of total soluble solids and in the second one, this juice was further concentrated in order to reach more than 600 g kg^{-1} soluble solids. The authors observed vitamin C losses of 2.5 and 3.1% during the first and the second stages, respectively.

The antioxidant activity of camu–camu juice did not increased in the same proportion that phenolics and vitamin C, which can indicate that some compounds present in the camu–camu juice, other than these ones, which also have antioxidant properties, may have been lost during processing.

Cassano et al. (2011) evaluated the concentration of pomegranate juice by ultrafiltration and osmotic evaporation and verified that the juice soluble solids content increased from 162 g kg^{-1} (clarified juice) to 520 g kg^{-1}. At the end of the process, anthocyanins and other biological active compounds of the juice were satisfactorily preserved.

Considering the coupling of both processes, the overall concentration factors achieved for total phenolics, vitamin C and antioxidant activity were 7.1, 5.9 and 7.3, respectively, while total solids increased 7.5 times. Therefore, the combination of reverse osmosis and osmotic evaporation can be considered a promising alternative for concentration of fruit juices, which can result in high quality products.

4. Conclusion

The coupling of reverse osmosis and osmotic evaporation processes showed to be a potential alternative to the concentration of camu–camu juice, resulting in a final product with 566 g kg^{-1} of solid content. Vitamin C was the compound that was most affected by the process conditions, due to its high sensitivity and instability. Phenolics were well preserved, with only 6% of losses along both processes. The juice concentrated by reverse osmosis and osmotic evaporation had vitamin C and total phenolic concentrations of 94.6 g kg^{-1} and 105.2 g kg^{-1}, respectively, showing to be a good source of bioactive compounds, which can be directly consumed or used in the formulation of other food products. However, an economic analysis is necessary in order to evaluate the viability of this process in an industrial scale.

Acknowledgements

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